

Performance Monitoring Protocol (QA/QC) for the Thermo TSQ Quantum GC/MS (EI/CI)

1 Scope

This document addresses the performance monitoring (QA/QC) of the Thermo TSQ Quantum GC/MS (EI/CI) System. This document applies to personnel using the associated instrument(s)/equipment in Quantico, VA in the following disciplines/categories of testing: Drug chemistry, toxicology, explosives (chemistry), fire debris, and Chemistry Unit general physical and chemical analysis.

2 Principle

The Thermo TSQ Quantum GC/MS consists of a TraceGC Gas Chromatograph (GC) and a Triple Stage Quadrupole (TSQ) Quantum Mass Spectrometer (MS). These two instruments work in tandem and are referred to as the TSQ Quantum. The instrument is configured with a combination electron impact (EI) ionization and chemical impact (CI) ionization source using an interchangeable ion volume system.

When the instrument is in EI mode, it is implied that an EI volume is being used and that the reagent gas is off. Alternatively, when in CI mode, it is implied that a CI volume is being used and the reagent gas is on. The instrument can also be used with a Solids Probe. Solids Probe is a sample introduction technique utilizing the mass spectrometer for analysis and can be in either EI or CI mode. Definitions and guidelines for following this protocol are outlined in the "General Instrument Maintenance Protocol."

3 Equipment/Materials/Reagents

- a. Instrumentation - Thermo TSQ Quantum, Thermo TraceGC Ultra, and data system with XCalibur software (or equivalent)
- b. Autosampler - CTC A200S or "Pal" Series automated sampler, accessories, and software (or equivalent)
- c. GC Column - Agilent DB-5MS, 30 m, 0.25 mm i.d., 0.25 μ m film (or equivalent)
- d. Carrier Gas - Helium, 99.99% (high purity)
- e. CI Reagent Gas - Methane, 99.99% (high purity)
- f. Chloroform, GC grade
- g. Lidocaine HCl (Sigma or equivalent)

- h. Tributoxyethyl Phosphate (TBEP) (Chem Service or equivalent)
- i. Perfluorotributylamine (PFTBA, FC-43) (Agilent or equivalent)
- j. Analytical balance
- k. Volumetric flask
- l. Autosampler vials - 2 mL GC vials, crimp or screw top, with or without 100-500 μ L inserts (Agilent or equivalent)
- m. Injection port liners - 3 mm split-splitless, tapered, with or without glass wool (Restek or equivalent)
- n. Injection port septa - low-bleed 17 mm (Restek or equivalent)
- o. Autosampler syringes - SGE Analytical gas tight 10 μ L (or equivalent)

4 Standards and Controls

4.1 Testmix (0.05 mg/mL each of Lidocaine and TBEP)

The testmix is used to assess daily operating performance, mass assignment, and continued integrity of the system. To prepare, weigh 5.8 mg Lidocaine HCl and 5 mg TBEP into a 100-mL volumetric flask. Bring to the mark with chloroform and mix well. Store the solution in the refrigerator. It has a shelf-life of three years. This preparation may be appropriately scaled up.

4.2 PFTBA Tuning Solution

The PFTBA tuning solution is used for tuning the mass spectrometer and verifying mass calibration. It is supplied by the instrument manufacturer and does not expire. It is stored in a glass container attached to the TSQ. Under normal conditions, this should not need to be refilled.

5 Calibration

Not applicable.

6 Sampling or Sample Selection

Not applicable.

7 Procedures

7.1 Daily Checks

The following steps will be performed daily. Enter the appropriate information in the QA/QC log for tracking purposes.

- a. Check to ensure that the GC wash vials are filled, the waste vials are empty, and all are in the appropriate positions.
- b. Record the remaining disk space on the hard drive. Use Windows Explorer or XCalibur to verify that the hard disk has at least 100 MB of free disk space. Do not use if less than 100 MB remain.
- c. Record the line pressure of the building helium supply (carrier gas). The regulator should read 50 p.s.i. or above. If it cannot maintain this pressure, contact appropriate instrument support personnel. If the helium is supplied by a gas cylinder, record the tank pressure. Change the tank if less than 100 p.s.i. remaining.
- d. If using CI mode, record the tank pressure of the methane tank (reagent gas). Change the tank if less than 100 p.s.i. remaining.
- e. Check the Ion Gauge to ensure that there are no significant leaks in the system. Do not use if the pressure is higher than 1×10^{-4} torr with the reagent gas off.
- f. Prepare instrument for EI mode or CI mode. In the Quantum Tune Master software, select the ionization mode under 'Setup.' Open both the corresponding tune and calibration files (such as EI_TUNE or PICI_TUNE). Insert the correct ion volume. Check that the reagent gas is ON with a value of 2.0 for CI mode and OFF for EI mode.
- g. Perform an analysis of the testmix. Open the appropriate testmix instrument method (such as 'TestmixEI' or 'TestmixCI'), and verify the parameters as listed in the 'Instrumental Conditions' section of this protocol. Set up a sequence, load the autosampler with a vial containing the testmix, and start the analysis. Evaluate the results using the 'Decision Criteria' section of this protocol. If the results are acceptable, print the following information for the testmix:
 - For EI: RIC of m/z 86, RIC of m/z 299, and TIC. Label the peaks with scan number and/or retention time.
 - For CI: RIC of m/z 235, RIC of m/z 399, and TIC. Label the peaks with scan number and/or retention time.
 - Complete mass spectrum of both Lidocaine and TBEP.

- h. If all requirements are within specification, prepare the documentation as outlined in the "General Instrument Maintenance Protocol." If any requirements fail, contact appropriate instrument support personnel.

7.2 As Needed Checks

The following steps will be performed as needed based on system performance. Indicate completion in the appropriate QA/QC log.

- a. Replace the septum in the GC injection port.
- b. Replace the liner within the GC injection port.
- c. Check the GC syringe in the autosampler. Replace if needed.
- d. Check the internal bungee cords in the autosampler. Replace if needed.
- e. Appropriate instrument support personnel or trained operator: Tune the mass spectrometer. Perform the following procedure for both EI and CI modes:
 1. Save the current tunes as backups with filenames such as 'EI_TUNE_BACKUP' or 'PICI_TUNE_BACKUP.'
 2. Perform a standard tune on both Q1 and Q3.
 3. Save the tune files when completed with filenames such as 'EI_TUNE' or 'PICI_TUNE'.
 4. Manual optimization of parameters may be performed to fine-tune the MS.
 5. Acquire a mass spectrum of the PFTBA for Q1 and Q3 in EI, Positive Ion CI, and Negative Ion CI ranging from 50 to 650 m/z:
 - For EI: Collect approximately 20 scans for each quadrupole under the filename "tunespec1".
 - For Positive Ion CI: Collect approximately 20 scans for each quadrupole under the filename "tunespec2".
 - For Negative Ion CI: Collect approximately 20 scans for each quadrupole under the filename "tunespec3".
 6. Evaluate the results using the 'Decision Criteria' section of this protocol. If the results are acceptable, print the mass spectrum from each.
- f. If all requirements are within specification, prepare the documentation as outlined in

the "General Instrument Maintenance Protocol." If any requirements fail, the IOSS Manager or appropriate instrument support personnel will determine the corrective maintenance to be taken.

8 Instrumental Conditions

8.1 Gas Chromatograph

Oven

Initial Temp: 60°C
Initial Time: 2.0 min
Ramp: 35°C/min
Final Temp: 250°C
Hold Time: 10.0 min

Inlet/Injector

Inj Vol: 1.0 µL
Mode: Splitless
Inlet Temp: 220°C

Column

Type: DB-5(MS)
Length: 30 m
Diameter: 0.25 mm
Film Thickness: 0.25 µm
Flow Mode: Constant Flow, Vacuum Compensation
Pressure: 1 mL/min
Carrier Gas: Helium

8.2 Mass Spectrometer

Solvent Delay: 5.0 min
Scan Mode: Full Scan
Scan Range: 50-500 m/z (EI)
100-500 m/z (CI)

Temperatures

Transfer Line: 280°C
Source: 185°C

9 Decision Criteria

9.1 Testmix

Verify the results of the testmix.

- a. In order for the instrument to be considered in good operating condition, both Lidocaine and TBEP should generate well-resolved, Gaussian-shaped peaks with baseline separation.
- b. A SNR of 3:1 will be the minimum response necessary to consider a response a peak.
- c. There should be no significant extraneous peaks in the chromatogram.
- d. The retention times of each component should be similar as compared to previous analyses (unless GC maintenance has been performed, such as column clipping or replacement).
- e. Check for the correct mass assignments for the mass spectra:
 - EI - Lidocaine ions 86 and 234 and TBEP ions 57, 199, and 299.
 - CI - Lidocaine ion 235 and TBEP ions 299 and 399.

9.2 Tune

Verify the results of the tune. Compare the results of the tune to previous tune results. The following are typical PFTBA values for the TSQ. If the observed PFTBA peaks are outside the values listed below, the IOSS Manager or appropriate instrument support personnel will determine the corrective maintenance to be performed.

- a. PFTBA Tune: Mass assignments for m/z 69, 219, 414, 502, and 614
- b. Relative abundance:
 - EI: 69 or 219 base peak, 414 and 502 present
 - CI+: 414 base peak, 219, 69, and 614 present
 - CI-: 633 or 452 base peak, 414 present

10 Calculations

Not applicable.

11 Measurement Uncertainty

Not applicable.

12 Limitations

Only properly trained personnel will perform duties involved in the operation, maintenance, or troubleshooting of this instrument.

13 Safety

Take standard precautions for the handling of all chemicals, reagents, and standards. Refer to the *FBI Laboratory Safety Manual* for the proper handling and disposal of all chemicals. Personal protective equipment should be used when handling any chemical and when performing any type of analysis. Many instrument components are held at temperatures of 250°C and higher. Precautions should be taken to prevent the contact of skin with heated surfaces and areas.

14 References

Manufacturer's Instrument Manuals for the specific models and accessories used.

"General Instrument Maintenance Protocol" (Inst 001) *Instrument Operation and Systems Support SOP Manual*.

"Gas Chromatograph General Maintenance Protocol" (Inst 002) *Instrument Operation and Systems Support SOP Manual*.

"Mass Spectrometer General Maintenance Protocol" (Inst 004) *Instrument Operation and Systems Support SOP Manual*.

FBI Laboratory Safety Manual.

Rev. #	Issue Date	History
0	06/21/06	New document which replaces original titled "Performance Monitoring Protocol (QA/QC) for the Finnigan TSQ GC/MS (EI/CI)."
1	05/01/08	Changed 'monthly' checks to 'as needed' in section 7.2. Corrected column information and instrument conditions in section 3 and 8.1. Updated ion ratios in section 9.2.
2	08/19/09	Updated with manufacturer's current name and model name (following replacement of outdated instrument with new model) in title and sections 1, 2, and 3a. Removed reference to old software in 7.1b. Updated steps for using new software in 7.1f. Added check of bungee cords in 7.2d. Removed reference to old software in 7.2e.iv. Changed final temp in 8.1 and transfer line temp in 8.2 to match other GC/MS instruments.
3	04/01/11	Updated manufacturer information on autosampler syringe in section 3. Changed PFTBA tuning decision ion for CI+ mode from m/z 619 to m/z 614 in section 9.2b.
4	10/04/18	Updated Section 1 Scope to include applicable disciplines/categories of testing. Updated heading in Section 6. Added 'appropriate instrument support personnel' to Sections 7.1 c & h, 7.2 e & f and 9.2. Updated Section 9.1 b & c to account for instrument variation and maintenance. Updated Section 14 and header to 'Instrument Operation and Systems Support.'

Approval

Redacted - Signatures on File


Drug Chemistry/
General Chemistry
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Date: 09/28/2018

Toxicology
Technical Leader:

Date: 09/28/2018

Fire Debris Technical
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 Date: 09/28/2018

Explosives (Chemistry)
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Date: 09/28/2018

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